SPUTTER TARGETS AND METHODS OF MANUFACTURING SAME TO REDUCE PARTICULATE EMISSION DURING SPUTTERING

Field of the Invention

5   The invention relates to sputter targets and methods of manufacture thereof that result in reduced particulate emission during sputtering.

Background

       Macroparticles are generated during sputtering by the presence of

10   dielectric inclusions in the metallic sputter target. Electrical charges build up on the dielectric particle and eventually electrical breakdown occurs. When this electrical breakdown occurs a high current density cathodic arc forms and melts a small section of the target. The arc pressure causes the liquid metal in the cathode to spurt out at high velocity and strike the intended substrate. The droplets solidify on the substrate

15   creating a large defect in the metallization layer.

       This problem has been frequently experienced in the sputtering of large flat panel displays that commonly operate at power densities of greater than 16 W/cm^2. The appearance of macroparticles on these flat panel displays causes gaps or shorts in the metallization patterns required for addressing the various pixels in the

20   display. Obviously, the gaps or shorts reduce the yield of acceptable display units.

       Sputtered aluminum layers are often the initial conductive layer that is coated on the substrate. In addition to the desire in having low inclusion levels in the Al target, the targets must also provide good resistance to hillock formation. It is known that alloying additions of silicon and copper are effective at reducing hillock

25   formation and that these alloying additions to aluminum do not significantly lower the electrical conductivity of the resulting sputtered Al films.

Summary of the Invention

       The invention centers around concurrent discoveries pertaining to (1)

30   the size of macroparticles that are especially deleterious in high power sputtering operations such as those employed in flat panel display sputtering operations and (2) improvements in filtering deleterious inclusions from the molten Al during target formation by the addition of small amounts of Si to the alloying mix.

Initially, it was discovered that metallic sputtering target materials with inclusions below 500 μm in size would perform substantially free from macroparticle defect generation at sputtering power densities up to about 20 W/cm². Further, sputter targets with inclusions below 400 μm in size were found to perform

5   substantially free of macroparticle generation at sputtering densities of about 25 - 30 W/cm². This is the sputtering power density range normally encountered in the sputtering of flat panel displays. At sputtering power densities of 40 W/cm² or greater, dielectric inclusions should be less than 300 μm in size in order to inhibit macroparticle contamination of the deposited film.

10   Further, during the casting process in which the molten metal, preferably Al, is cast into a sputter target. It has been found that addition of a small amount of Si into the molten metal improves the filtering performance of the ceramic filter element used in the semi-continuous casting method to thereby help to separate the undesirable inclusions from the molten mass before it is solidified.

15   After the sputter target with reduced inclusions has been prepared, it is tested to ascertain the amount and size of inclusions contained therein. When the target is adapted to sputter coat flat panel displays and the like wherein sputtering power densities of greater than about 25 W/cm² are commonly encountered, targets are rejected that contain measurable quantities of inclusions having sizes on the order

20   of 400 μm and greater.

Two test methods are in common use today to detect size and quantity of defects in the metal. In the more prevalent ultrasonic method, the Al alloy ingot is immersed in a water tank while a 10 to 15 MHz ultrasonic transducer scans the part. The ultrasonic pulse is able to detect defects in the metal. An inclusion or void in the

25   metal reflects the ultrasonic energy and is detected. Histograms, calibration charts and statistical calculations can be made using the reflected signals to determine the overall quality of the metal.

In the less common dissolution method, a small sample of the aluminum alloy is dissolved in acid and the acid solution is passed through a fine

30   (typically 5 μm pore size) filter. The filter is then examined using optical and scanning electron microscopes to determine the number, composition and size distribution of inclusions in the metal sample.

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The invention will be further described in the following detailed
description which is to be read in conjunction with the appended drawings.

Brief Description of the Drawings

5   Fig. 1 is a graph showing the correlation between inclusion sizes and
macroparticles sputtered from an Al 0.5 Cu target at a power density of 25 W/cm^2;

Fig. 2 is a graph showing the reduction in inclusions in targets
wherein Si has been added as a filtering aid during the Al casting process;

Fig. 3 is a graph comparing surface tension for Al-Si and Al-Cu melts;

10   Fig. 4 is a schematic diagram of a semi-continuous casting process
used to cast reduced inclusion containing Al targets in accordance with the invention;

Fig. 5 is a schematic diagram illustrating a preferred method for
assessing target "cleanliness" in accordance with the invention; and

Fig. 6 is a schematic block form illustration of a test system for

15   assessing the "cleanliness" of targets.

Detailed Description of the Preferred Embodiment

We have discovered that the cause of macroparticle generation during
sputtering is the presence of relatively large dielectric inclusions in the metal

20   sputtering target. Measurements of the relationship between inclusion size and
macroparticle generation have shown that the generation of macroparticles depends
upon the size of the inclusion. At a sputtering power of 25 W/cm^2, inclusions with
size greater than around 400 μm are required to produce arcs of sufficient intensity to
generate macroparticles. Inclusions with surface areas below this value can generate

25   arcs, but the intensity of the arcs is not sufficient to melt the aluminum target spot
and provide a large enough pressure wave to eject molten metal macroparticle
droplets. Above this threshold inclusion surface area, the arcs have sufficient energy
to melt a small spot on the target and eject molten metal droplets from the target.
Fig. 1 shows how the number of molten metal macroparticle droplets increases with

30   inclusion size in a test sputter system having a sputtering power density of 25 W/cm^2.

Accordingly, one way to reduce the number of macroparticles
generated during aluminum target sputtering is to reduce the number of large

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dielectric inclusions contained in the metal target. In addition to low dielectric

inclusion content, the target needs to provide an aluminum film on the substrate that

has good electrical conductivity and good resistance to hillock formation. It is well

known that alloying additions of silicon and copper are effective at reducing hillock

5   formation and that these alloying additions to aluminum do not significantly lower

the electrical conductivity of the sputtered aluminum films. However, it was not

known until our work that the addition of silicon significantly reduces the dielectric

inclusion density in aluminum alloys. This lower propensity to generate

macroparticles is the result of alloying the aluminum target material with silicon.

10   The silicon lowers the macroparticle generation by significantly improving the

efficiency of aluminum filtering during sputtering target manufacture.

        One aspect of the invention relates to aluminum alloy sputtering

targets that contain a small amount of silicon so that the hillock formation is reduced

in the film while the electrical conductivity of the film is not significantly reduced

15   from that of pure aluminum. Additionally, the propensity for macroparticle

generation during sputtering is greatly reduced. Fig. 2 shows results of a series of

measurement on sputtering targets manufactured with varying amounts of copper and

silicon alloy additions. A significant reduction in dielectric inclusion concentration

is found when silicon is added to the alloy. This result is unexpected. The

20   explanation for this result is that the addition of silicon to the aluminum melt

changes the aluminum alloy surface tension so that the aluminum metal efficiently

wets the ceramic filter used to remove inclusions from the molten aluminum during

casting. In this way the dielectric inclusion level in the cast metal is reduced and the

generation of macroparticles from the target is eliminated.

25   Table 1 provides detailed inclusion measurements for various Al-Si-

Cu alloys. It is clear from the data in Table 1 and Fig. 2 that the addition to the

aluminum melt of ever small amounts of silicon (-0.2 wt% silicon) has a significant

effect on lowering the number of dielectric inclusions in the aluminum. Adding

copper to the melt appears to reduce the inclusion levels to a small extent, but silicon

30   is significantly more effective.

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TABLE 1

Alloy (wt%)	Inclusion Count AVG	Inclusion Count STDEV
Al-0.5Cu	88	96
Al-1Cu	43	8
Al-0.2Si-0.5Cu	2	3
Al-0.75Si-0.5Cu	8	1
Al-1Si-0.5Cu	7	9
Al-1Si-1Cu	19	17
Al-1Si	28	9

Table 1. Inclusion counts in aluminum alloy sputtering targets.

The mechanism by which silicon reduces the dielectric inclusion level in the target is thought to be by significantly improving the effectiveness of the ceramic filter in removing dielectric inclusions from the melt during casting. Fig. 3 shows the surface tension for Al-Si and Al-Cu melts.

Since alloying additions usually reduce the electrical conductivity of metals, it is important to use as little alloying addition as is required to achieve desired characteristic. Consequently, the preferred alloy for this invention is Al-0.5Si-0.5Cu. The copper addition is useful for reducing hillock formation in the deposited film, while the silicon addition is useful for reducing dielectric inclusions in the aluminum alloy target. Copper may be added to the alloy in an amount of about 0.01 - 3.00 wt%.

In accordance with the process used in the invention to inhibit the amount of undesirable inclusion in sputter targets, high purity metal, such as aluminum or aluminum alloy is first melted. From about 0.01 to 2.00 wt% of Si is added to the melt.

Although applicants are not to be bound to any particular theory of operation, it is thought that addition of a small amount of Si into the melt decreases

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the surface tension of the melt so that the metal, such as Al, will wet the filter better. This, in turn, enhances the efficiency of the filter used in the casting system so that the inclusions better adhere to the filter during processing.

5   Turning now to Fig. 4, the method for forming ingots of the desired metal, preferably Al and Al alloys, is schematically illustrated.

The metal, any alloying element, and Si are admitted to the induction melting furnace 101. The components are melted and degassed in injecting a sparging mixture of high purity argon and trace chlorine. The gas is injected thru a spinning nozzle 102 to shear the bas bubbles to very small size to provide greater
10   surface area contact; thereby increasing the efficiency of the gas.

Degassing is performed for a period of time prior to casting. The dross is skimmed from the top and then the degassing is continued during the casting process. The degassing treatment helps to clean the metal of both dissolved gasses and suspended impurities. It also reduces unwanted alkali elements and Mg.

15   To initiate the cast, the induction furnace is pressurized using the sparging gas described above, with some additional argon. While closely monitoring metal and system temperatures, the metal is forced out of the furnace via a graphite pipe 103. This is a unique aspect of the invention in that it protects the metal from air contact and turbulence until it reaches the filter box.

20   The melt is then delivered to a preheated filter box 104 containing a sintered ceramic filter medium 175. Preferably, the filter is a sintered alumina filter of the type available from Mettaullics System Co., L.P., Solon, Ohio. The particular filter element presently preferred is an 8-grit porosity which equates to about 40 - 50 ppi (pores per linear inch).

25   Although the sintered alumina is presently preferred when Al metals and alloys are filtered, other ceramics such as Si, C, Ti, B_2, B_4, C, SI_3, N_4, Si Al ON and zirconia can also be mentioned as exemplary filter components. Filters of the type that may be employed are described in U.S. Patent 5,369,063, (Gee et al.), the disclosure of which is incorporated by reference therein.

30   Passage of the Si containing melt through the filter element removes a significant number of the inclusions that were not removed in the degassing process. After passing through the filter element, the metal is fed to a distribution trough 105

which evenly distributes and feeds metal to each of the water cooled ingot casting molds 107, 108. During feeding into the molds through the insulated hot top, the metal is vibrated (stirred) via low frequency inductive stirring coils 116, 118 to enhance the grain structure of the ingots.

5   The molten metal is fed and solidified at a controlled casting rate via contact with the water cooled molds (primary cooling) from which emerge the high quality solidified ingots 190, 192. The casting speed is controlled using computer-controlled hydraulic casting equipment 180. The ingot structure and surface quality are enhanced by advanced secondary cooling provided by the mold cooling water

10   exiting the water-cooled mold and coming into direct contact with the solidified ingot as it emerges from the mold (the vertical direct chill casting process).

In the case of casting of Al and Al alloy, such as Al 0.5% Cu ingots, the ingot is saw cut into the desired target blank size. Then it is pressed and rolled. The blank is then machined, bonding of the target to an Al 6061 backing plate may

15   preferably be conducted as reported in patent applications WO 00/15863 and PCT/US00/18968 filed July 12, 2000. The bonded target/backing plate assembly is then ready for usage in conventional cathodic sputter coating operations preferably those adapted for sputter coating of flat panel displays.

The sputter targets made in accordance with the above process have

20   exhibited reduction in macroparticle generation particularly in high power systems such as those sputter systems employed to coat thin layers on flat panel displays. Such systems commonly operate at high power densities of about 25 W/cm^2 and greater and, in the past have been plagued by poor sputtering uniformity due to macroparticle generation during sputtering.

25   For example, Al and Al targets made by the above process and containing reduced inclusion levels can be used in sputtering processes to form an Al layer in flat panels of the type described in U.S. Patent 6,019,657. As is typical in such structures, the sputter coated Al layer is then coated with a cladding layer such as would be formed with Mo, W, Ti, Nb, Ni, Cr, metal sillicides etc. The flat panel

30   displays are used in liquid crystal, electro-luminescent, gas plasma and vacuum fluorescent displays.

The Al targets are sputtered using conventionally available sputtering

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equipment of the type functioning to coat large flat panel displays. In such systems, the target is attached to the cathode and a power density of at least 25 W/cm^2 is imparted to the system, resulting in ionic bombardment of the target from the charged plasma cloud in the apparatus above the target. The ionic bombardment of

5   the target dislodges the desired coating material from the target surface and this material is deposited on the flat panel positioned proximate the anode.

In order to test the targets manufactured by the above process to ensure that inclusion content is reduced, an ultrasonic scanning method is preferably used. Equipment and methods necessary to scan for target inclusions can be seen in

10   U.S. Patent 5,406,850 of common assignment herewith. This disclosure is incorporated by reference herein.

In accordance with the test, target inclusions seen by the scanning process are compared against calibration values. If inclusions are too large, the target is rejected. Scanning can be conducted in both the "race track" and remaining

15   portion of the sputter surface. It is presently preferred to reject targets wherein inclusions of greater than 400 μm are noticed in the "race track." Inclusions of less than 800 μm may be tolerated in the remaining non-race track areas.

Another non-destructive method for characterizing a sputter target material and comprises the steps of sequentially irradiating a test sample of the

20   sputter target material with sonic energy at a plurality irradiating on a surface of the sample; detecting echoes induced by the sonic energy; discriminating texture-related back scattering noise from the echoes to obtain non-rectified radio frequency echo waveform signals; monitoring non-rectified echo waveform signals for the 180 degree waveform phase inversion, comparing the non-rectified echo waveform

25   signals with said at least one of each: phase inverting and phase non-inverting calibration values, to detect void-like and particle-like flaw data points separately and no-flaw data points; counting the flaw data points for the each flaw type separately as well as all together to determine a total flaw count $C_{FT \text{ (without phase inversion)}}$, counting the flaw data points and the no-flaw data points to determine a total number of data

30   points C_{DP}; and calculating a total cleanliness factor $F_{CT} = (C_{FT} / C_{DP}) \times 10^6$ as well as cleanliness factors $F_{CI} = (C_{FI} / C_{DP}) \times 10^6$ and $F_C = (C_{FN} / C_{DP}) \times 10^6$ for each sort of flaws separately. Cleanliness factor C_{FI} represents phase inverted flaws such as

voids contained in the metal. C_{FN} represents non-phase inverted flaws such as inclusions in the metal.

Unlike many prior art methods, this test method provides a characterization of the sputter target material by separate identification of and

5   counting of void-like and particle-like (i.e., inclusion) flaws. A partition of cleanliness factor for components associated with different kinds of flaws defines the rejection criteria more precisely by identifying and sizing the flaws of different kind.

Although the cleanliness factor provides a useful characterization of the sputter target material, quality control information can also be provided by means

10   of a histogram. More specifically, the sputter target may be characterized by defining a plurality of amplitude bands for each type (waveform inverting and non-inverting) of flaws; measuring said modified amplitude signals to determine modified amplitude signal magnitudes; comparing said modified amplitude signal magnitudes with said plurality of amplitude bands to form subsets of said modified amplitude

15   signals; counting said subsets of modified amplitude signals to determine a plurality of modified amplitude signal counts, each modified amplitude signal count of said plurality of amplitude signal counts corresponding to one of said amplitude bands of said plurality of amplitude bands; and constructing a pareto histogram, combining individual histograms for both flaw classes, relating said modified signals counts to

20   said plurality of amplitude bands.

These methods for characterizing sputter target materials may be used in processes for manufacturing sputter targets. The cleanliness of a sputter target and particularly cleanliness from non-phase inverting flaws is the primary factor determining the quality of layers or films produced by the target. By shaping only

25   those sputter target blanks having cleanliness factors or histograms meeting certain reference criteria to form sputter targets, and rejecting blanks not meeting those criteria, one improves the likelihood that the sputter targets so manufactured will produce high quality layers or films.

Fig. 5 illustrates one method for sorting of flaws and characterizing

30   the cleanliness of sputter target material. In accordance with this method, a cylindrical sample of the sputter target material (which preferably comprises metal or a metal alloy) is compressed or worked to produce a disc-shaped test sample 52

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having a planar upper surface 54 and a planar lower surface 56 approximately

parallel to the upper surface 54. The transducer 60 irradiates the upper surface 54 of

the test sample 52 with a single, short-duration, megahertz frequency range ultrasonic

pulse 62. The transducer 60 subsequently detects an echo 64 induced in the test

5   sample 52 by the pulse 62. The transducer 60 converts the echo into an electrical

radio frequency signal (not shown), which is processed to retrieve the waveform

phase and maximum amplitude information.

More specifically, the sample first is compressed along a dimension to

form the disc-shaped test sample 52. Preferable, the sample is compressed by

10   forging or rolling, followed by diamond cutting to prepare the planar surfaces 54 and

56. The reduction in the dimension may be anywhere between 0% to 100%. The

compression of the sample flattens and widens certain flaws. The target comprises

both void-like defects 91 and inclusions or undesirable particles 93.

As illustrated in Fig. 6, the test sample 52 then is immersed in

15   deionized water (not shown) in a conventional immersion tank 80. The transducer

60 is mounted on a mechanical X-Y scanner 82 in electrical communication with a

controller 84 such as a PC controller. The controller 84 is programmed in a

conventional manner to induce the mechanical X-Y scanning unit 82 to move the

transducer 60 in a raster-like stepwise motion across the upper surface 54 of the test

20   sample 52.

The transducer 60 is sold by ULTRAN USA under the designation

WS50-10P4.5. This is a long focal length piezoelectric transducer having a fixed

focal length of 114 mm (in water). At a peak frequency of approximately 9.15 MHz

with 8 MHz (-6 dB) bandwidth, the transducer produces a pulse 62 having a focal

25   zone (-6 dB) of approximately 21 mm in aluminum and a focal spot 0.8 - 0.9 mm in

diameter.

Most preferably, the upper surface 54 of the sample 52 has a width or

diameter on the order of approximately 28 cm. Data acquisition steps of

approximately 0.9 mm in both the "x"-direction and the "y"-directions permit the

30   detection of 0.25 mm flat bottom holes at a detection level of -6 dB without exposure

area overlap. One thereby irradiates approximately 100,000 test points on the upper

surface 54.

Most preferably, the transducer 60 is oriented so that the pulse 62 propagates through the deionized water (not shown) in the immersion tank 80 and strikes the test sample 52 approximately normally to the upper surface 54. Furthermore, the transducer 60 is preferably spaced from the upper surface 54 such

5   that the pulse 62 is focused on a zone 86 (Fig. 2) of the test sample 52 between approximately 3 mm and 23 mm below the upper surface 54. The pulse 62 interacts with the sample 52 to induce echoes 64, which then propagate back through the deionized water (not shown) to the transducer 60 approximately 60 μsec after the pulse is sent.

10   An especially preferred echo acquisition system includes a low noise receiver comprising a low noise gated preamplifier 90; a low noise linear amplifier 92 with a set of calibrated attenuators, a 12-bit (2.44 mV/bit) analog-to-digital converter 94 and digital oscilloscope 95 connected with receiver analog output. When sufficient time has elapsed for the echoes to arrive at the transducer 60, the

15   controller 84 switches the transducer 60 from a transmitting mode to a gated electronic receiving mode. The echoes 64 are received by the transducer 60 and converted into an RF electric signal (not shown). The RF signal is amplified by the preamplifier 90 and by the low noise amplifier 92 to produce modified amplitude signal and displayed on the screen of oscilloscope 95 to extract waveform phase

20   information. The modified amplitude signal then is digitized by the analog-to-digital converter 94 before moving on to the controller 84. The analog-to-digital conversion is performed so as to preserve amplitude information from the analog modified amplitude signal.

Flaws of given nature (void-like or alumina inclusions) are

25   determined by monitoring for waveform phase inversion using digital oscilloscope 95. Flaws of given sizes are detected by comparing the digitized modified amplitude signals obtained from the sample 52 with reference values derived from tests conducted on reference samples (not shown) having composition similar to that of the test sample 10 and either blind, flat-bottomed hole of fixed depth and diameter or

30   alumina particles of given size artificially imbedded into reference sample material.

The especially-preferred PC controller 84 controls the data acquisition process. An especially-preferred software package used in connection with the data

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acquisition system is available from Structural Diagnostics, Inc. under the designation SDI-5311 Winscan.

The PC controller 84 is also programmed to calculate the total cleanliness factor and the cleanliness factors for the sorted flaws characterizing the

5   material of the sample 52. More precisely, it is programmed to discriminate texture-related back scattering noise and to distinguish "void-like flaw data points from the alumina particle-like flaw data points". The PC controller 84 maintains a count of the flaw data points detected during the testing of a test sample 52 to determine a flaw count "C_{FT}", "C_{FI}", "C_{FN}".

10   The PC controller 84 also is programmed to distinguish "no-flaw data points," that is, digitized modified amplitude signals representing amplitudes which, after comparison with the calibration values, indicate the absence of flaws.

The PC controller also determines a total number of data points "C_{DP}," that is, the sum of the flaw count C_{FT} and the number of no-flaw data points.

15   Although the total number of data points could be determined by maintaining counts of the flaw data points and the no-flaw data points, it is preferably determined by counting the total number of positions "C_I" along the upper surface 54 at which the test sample 52 is irradiated by the transducer 60 and subtracting the number of digitized RF signals "C_N" which the data acquisition circuitry was unable, due to

20   noise or other causes, to identify as either flaw data points or no-flaw data points. (Alternatively, the "noise count" C_N may be described as the number of positions along the upper surface 54 at which neither a flaw data point nor a no-flaw data point is detected.)

Having determined the flaw counts C_{FT}, C_{FI}, C_{FN}, and the total number

25   of data points C_{DP}, the PC controller is programmed to calculate the cleanliness factor $F_{CT} = (C_{FT} / C_{DP}) \times 10^6$, $F_{CI} = (C_{FI} / C_{DP}) \times 10^6$, $F_{CN} = (C_{FN} / C_{DP}) \times 10^6$ to characterize the material comprising the sample 52. Unlike the prior art "flaws per cubic centimeter," the magnitude of the cleanliness factor is not dependent on any estimate of pulse cross-sectional area. Since the cleanliness factor is normalized by

30   the dimensionless coefficient $C_{DP} \times 10^{-6}$ rather than by volume, it is more closely related to ppm and ppb units than are units of "flaws per cubic centimeter."

The preparation of a suitable program for determining the cleanliness

factor in accordance with the invention as disclosed herein is within the ordinary skill in the art and requires no undue experimentation.

Another way in which to characterize the material comprising the sample 52 is by determining the size distribution of flaws in the test sample 52.

5   More specifically, one may characterize the cleanliness of the sample 52 by defining amplitude bands or ranges; comparing the amplitudes represented by the digitized modified amplitude signal for certain types of flaws (phase inverting and phase non-inverting) with the amplitude bands to form subsets of the modified amplitude signals; counting these subsets of modified amplitude band and for each type of

10   flaws; and constructing a pareto histogram relating the modified signal counts to said plurality of amplitude bands. Since the amplitudes represented by the digitized modified amplitude signals for each type of flaws are related to the sizes of flaws detected in the sample 52, the histogram provides and indication of the flaw size distribution in the sample 52.

15   The preparation of a suitable program for plotting histograms herein is within the ordinary skill in the art and requires no undue experimentation.

Either the cleanliness factor or histograms may be used as a product quality test. As noted earlier, the cleanliness of a sputter target is one factor determining the quality of the layers or films produced by the target. By shaping

20   only those sputter target blanks having cleanliness factors and particularly C_{FN} less than reference cleanliness factors, or having histograms with selected columns or areas less than reference values, to form sputter targets, and rejecting blanks not meeting those criteria, one improves the likelihood that the sputter targets so manufactured will produce high quality layers or films.

25   While the form of apparatus herein described constitutes a preferred embodiment of this invention, it is to be understood that the invention is not limited to this precise form of apparatus, and that changes may be made therein without departing from the scope of the invention which is defined in the appended claims.

What is claimed is:

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